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(54) **Insulation system.**

(57) A method and apparatus for directing a stream of bulk fibers while simultaneously spraying an inorganic temperature resistant tacky binder material into the stream of fibers. A layer of high temperature insulating fibers may thus be formed. The binder may be a phosphate tackifying agent mixture or a colloidal suspension of materials with the tackifying agent. The tackifying agent is preferably montmorillonite clay.

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Background of the InventionField of the Invention

The invention relates to a method for applying a layer of  
5 fibers coated with a tacky inorganic binder onto a surface. More  
particularly, the invention is a method for spraying a layer of  
refractory fibers coated with a high temperature inorganic binder  
including a montmorillonite clay. By way of further  
characterization, but not by way of limitation thereto, the  
10 invention is a method and apparatus for spraying refractory fibers  
coated with a novel binder including a montmorillonite clay onto a  
surface and curing the coated fibers.

Description of the Prior Art

In the past, high temperature resistant fibrous material  
15 has been applied to heated surfaces, such as heat treating furnaces  
and kilns, by attaching batts or strips containing such fibers to  
the heated surface. This method requires mechanical anchoring or  
fastening means to attach the strips to the heated surface. Such  
mechanical attachment is expensive in that it requires large amounts  
20 of labor and fastening materials to attach the strips. In addition,  
because this method is time consuming, it requires the furnace or  
kiln to be out of service for extended periods of time. Finally,  
gaps between adjoining strips may occur either during the attachment  
process or later due to thermal shrinkage which allows heat leakage  
25 between the layers of fiber.

Improvements to this method of attaching refractory fiber  
linings to the wall of kilns and furnaces have been made such as is  
described in U. S. Patent No. 4,381,634. This method involving the  
mechanical attachment of modules, overcomes many of the problems  
30 associated with layered linings especially the opening of gaps  
between the edges of layers due to heat shrinkage at elevated  
temperatures. However, like the above method, the use of modules  
requires mechanical attachment to the metal shells of furnaces which  
again require considerable cost in labor. The use of modules has  
35 also been found to be of value in relining old dense refractory  
linings such as refractory brick. In this case the folded or  
pleated modules are glued to the old monolithic or brick lining by

5 use of a refractory mortar or glue which is generally of the hydraulic setting nature. This method of relining of kilns is labor intensive requiring the application of mortar to the brick work and then quickly applying the folded modules thereon. This method requires that time be given for the setting of the hydraulic mortar before the newly lined kiln can be fired.

10 All the above methods have one feature in common which is that they present an unbonded refractory fiber lining to the interior of the kiln. As time goes by these fibers devitrify (crystallize) and in so doing lose some of their mechanical strength making them more susceptible to mechanical abrasion and destruction due to high velocity gases from the heating mechanisms of the kilns.

15 High temperature resistant binder powders have been mixed with water and sprayed with refractory fibers. Such methods typically employ calcium alumina cements which are very alkaline and will settle when in suspension. It is also difficult to achieve homogenous mixing of fiber and binder with such a system. A hydraulic bond must be achieved prior to drying of the cement otherwise bonding of the fibers will not occur. In addition, 20 careful drying of the sprayed mixture must be undertaken in order to prevent violent spalling.

25 Other methods for spraying insulation have been employed in the past utilizing organic binders. For example, U.S. patent 2,929,436 issued on March 22, 1960 discloses a method and apparatus for spraying a mixture of fibers and resin material. As indicative of prior methods of application, this patent discloses mixing fiber and resin in a nozzle and spraying the mixture onto a surface. However, while suited for its intended purpose, such method and apparatus, if used in the spraying of refractory fiber with a tacky 30 inorganic binder, would cause clogging of the nozzle. In addition, incomplete coating of the refractory fibers could cause a weakened layer of fibers which in turn could cause failure of the insulating layer at high temperatures. Because of the temperatures to which refractory fibers are subjected, often 2,000°F or more, such 35 failures could be dangerous to workers in such industries as the steel industry and could cause extensive damage to furnaces and kilns. Thus, the intimate admixture of refractory fiber and the

tacky inorganic binder to form a monolithic layer is important to the safe and efficient operation of such furnaces and kilns.

Prior art binders for bonding refractory fibers have included a variety of compounds including colloidal silica and phosphoric acid (actually  $P_2O_5$  which may be derived from  $H_3PO_4$  under appropriate temperature conditions). While these binder systems are useful in certain applications, they are not without relative disadvantages. For example, prior art phosphoric acid binders do not adhere well to refractory fibers. Binders including colloidal silica are subject to irreversible precipitation of the silica if the binder has been subjected to freezing temperatures.

A very serious disadvantage with prior art binder systems is binder migration. That is, as the binders are cured, the binder migrates to the surface of the material producing a material with a very soft interior. This causes serious integrity problems with the resulting material. The material is thus not suitable for use in applications requiring a monolithic structure. In addition, prior binder systems employing colloidal suspensions such as colloidal silica or alumina must be cured very carefully. That is, because of binder migration, the colloidal sol must be allowed to gel prior to curing. Failure to do so results in migration of the binder particles from the interior to the surface of the insulation layer. In addition, prior art binders must be sprayed onto a cool surface thus necessitating relatively long cooling periods before a furnace can be insulated.

Another disadvantage of prior art binders has been that they are not nearly as temperature resistant as the refractory fibers themselves. Thus, high temperature applications of the fiber-binder mix are limited by the properties of the binder rather than the fiber. For example, an insulation product made from refractory fibers which are themselves temperature resistant up to 2600°F may only be applied in temperature environments up to 2000°F if the binder used in that product is only effective up to 2000°F. Any reaction of the binder and fiber which tends to flux the fiber composition causes increased fiber shrinkage. Thus, the use temperature of the fiber - binder system is limited to a temperature less than the use temperature of the fibers alone.

Other practical problems encountered with the use of prior binder systems is that they are expensive, there may be safety and environmental problems in their applications, they may lack good suspension properties, and finally, as with prior phosphoric acid binders, the binders do not possess the tackiness or adhesiveness properties which are desirable in many applications. In applications where it is desirable to spray the refractory fiber onto a surface such as a furnace wall, it is essential that the binder be sufficiently tacky to cause the fibers to adhere to one another and to the surface.

Summary of the Invention

The invention is a method for applying a layer of fiber to a surface while simultaneously coating the fiber with a tacky inorganic binder. The layer of binder coated fiber is cured such that the layer becomes monolithic. The fibers are coated with the binder as they are directed from the spray gun and the coated fibers are caused to stick to each other and to the surface toward which they have been directed. Once the fibers have been bonded to the surface, they may be compressed by means such as tamping in order to alter the density of the layer. The layer is cured such that all of the moisture is driven out of the layer. Preferably, the layer is subjected to a temperature of at least 350°F for a few hours in order to cure the binder and fiber. However, the layer can be immediately exposed to temperatures as high as 1000°F without damage.

The present invention combines an inorganic binder matrix with temperature resistant clays. The tacky nature of the binder renders it suitable for a wide variety of applications. The resulting binder of the present invention is temperature resistant to at least 3000°F which allows its use with high temperature refractory fibers. As used herein, refractory fibers are defined as inorganic amorphous or crystalline fibers which are not adversely affected by temperatures in excess of 1500°F. An example of such fibers is alumina-silica fibers. However, as is known in the art, fibers including zirconia, chromium, calcium, magnesium and the like may be utilized. The binder disclosed herein could also be employed with metal oxides to provide an insulating layer of such material.

In addition, the binder system of the present invention greatly reduces or eliminates binder migration which occurs in prior art binders.

5 The binder includes a tackifying agent in combination with a phosphate binder matrix such as aluminum phosphate. The preferred tackifying agents are temperature resistant clays such as montmorillonite clays. The most preferred clay is western bentonite clay. A fluxing agent may also be added to the binder in small quantities to increase the strength of the binder to fiber bonds at  
10 elevated temperatures. Chromium oxide may also be added to the binder to improve the stability of the binder to fiber bonds at elevated temperatures. In addition, chromium oxide adds color to the binder which may be advantageous in the coating process.

15 In addition to phosphate compounds, colloidal suspensions such as colloidal silica, colloidal alumina, and colloidal zirconia may be combined with the tackifying agents disclosed herein to produce a suitable binder. When the disclosed tackifying agents are combined with these colloidal suspensions the severe binder migration problems associated with colloidal binders such as  
20 colloidal silica are overcome. The addition of the tackifying agent imparts good suspension characteristics to the binder. Additionally, the preferred binder of the present invention may be sprayed directly onto hot surfaces thus eliminating long delays in  
25 insulating furnaces requiring complete cool down. Finally, the novel binder claimed herein reduces shrinkage of the coated fibers thus improving their refractory characteristics. Because shrinkage is minimized, failure of the insulation layer due to cracks in the  
30 layer is reduced. Prior art binders, especially colloidal silica binders without the tackifying agent disclosed herein required gelling of the colloidal silica. Otherwise, serious binder migration to the surface of the insulation layer would occur.

35 Brief Description of the Drawings

Fig. 1 is an illustration of the binder coated fiber being applied to a furnace;

Fig. 2 is an enlarged perspective view of a novel spray gun for carrying out the method of the present invention;

Fig. 3 is a vertical cross-sectional view taken along line 3-3 of Fig. 2;

5 Fig. 4 is an elevational view of the novel spray gun;

Fig. 5 is a front perspective view of an alternate embodiment of the spray gun providing a circular fiber path; and

Fig. 6 is a side sectional view of a preferred spray gun for carrying out the method of the invention.

10 Description of the Preferred Embodiment

Referring to Fig. 1, an operator 11, dressed in suitable protective clothing 12 including an enclosed breathing apparatus 13, is shown holding a spraying apparatus generally designated as 14 from which a stream 15 of binder coated refractory fiber is being  
15 directed onto furnace wall 16. As used herein, refractory fibers are defined as inorganic, amorphous, or crystalline fibers which are not adversely affected by temperatures in excess of 1500°F. Examples of such fibers are alumina-silica-zirconia fibers, alumina silica fibers and other refractory fibers as are known in the art.  
20 Preferably the fiber is blown by air pressure from a suitable compressing apparatus (not shown). Furnace wall 16 is a bare metal wall of furnace 11. Similarly, ceiling 17 is a bare metal wall of furnace 11 onto which stream 15 may be directed. A previously fired or new refractory brick wall 18 of furnace 11 may also be coated  
25 with the layer of binder coated fiber. Similarly, a wall 19 which has been previously covered with refractory fiber modules as is known to one skilled in the art may also be coated utilizing the method of the present invention. A second operator, generally designated 21, also in protective clothing and breathing apparatus,  
30 is shown with a compressing apparatus 22 which may be used for tamping the layer of binder coated fiber so as to alter the density of the layer deposited by the method of the present invention.

Referring to Fig. 2, spray gun 14 is shown in more detail. More specifically, the refractory fiber stream 24 is directed  
35 through conduit 23 so as to emerge from an annular path defined by a deflector body 25 and a manifold 26. Deflector body 25 is preferably cone shaped and is supported in a manifold 26 by one or

more vanes 27 as illustrated in Fig. 3. Deflector body 25 deflects the flow of bulk fibers so as to assume an annular path in an oval or circular configuration depending upon the shape of deflector 25 and manifold 26. Thus a thin ribbon flow of fibers exits in an annular path to be coated on both sides as explained below.

The binder solution is fed through a binder feed line 27 and binder valve 28 into an external nozzle manifold 26 which feeds a plurality of spaced nozzles 31 surrounding the outside 37 of the annular fiber stream 24. Nozzles 31 are directed inwardly so as to spray the liquid binder solution onto the outer fibers 37 as they emanate from spray gun 14. Likewise, a ring of internal nozzles 32 are provided on deflector 25 so as to spray binder into the inner surface 30 of fiber stream 24. Binder is fed under pressure to nozzles 31 through binder feed line 28, valve 29 and manifold 26. Binder is fed under pressure to nozzles 32 through line 28, valve 29 and deflector 25. The fibers are uniformly wetted by the binder from two sides as they are being blown from the gun 14. Thus, the fibers are uniformly coated with liquid binder solution before reaching the surface to be coated.

Referring to Fig. 4, a side elevational view of spray gun 14 is shown as connected to flexible tubing 33. Flexible tubing 33 allows the fiber to be fed therethrough to conduit 23 in the spray gun 14 from a remote location thereby allowing the operator freedom to move about the area to be sprayed. Similarly, binder is fed to gun 14 through flexible line 28.

Referring to Fig. 5, a perspective view of an alternate embodiment of the spray gun is shown in which the deflector 25 and manifold 26 are circular such that annular path 24 of fibers being blown from the spray gun 14 is circular.

Referring to Fig. 6, a side sectional view of a preferred embodiment of a novel spray gun is shown. In this spray gun, no deflector is used to alter the path of fiber stream 24. Rather a nozzle 36 connected to, and supported by, a feed line 35 located in the approximate center of the path of fiber stream 24. Thus while nozzles 31 spray binder into the outer portions 37 of fiber stream 24, nozzle 36 ensures that the fibers in the center of fiber stream 24 are coated with binder. Nozzle 36 sprays binder in a conical



path as shown in order that the fibers be completely coated. It should be expressly understood that, while one nozzle 36 is disclosed, it is within the full intended scope of the invention that more than one nozzle 36 may be located in fiber stream 24.

5           Thus, the combination of nozzles 31 and 32 serve to uniformly coat the fibers in moving stream of fibers 36. As such, all of the fibers are coated with the tacky inorganic binder. This is especially important where the binder is extremely tacky and thus is difficult to spray into a moving stream of material to any  
10 depth. Because the fiber and binder are being used in a high temperature environment it is essential that the fibers be coated uniformly in order that the layer formed by the coated fibers be of sufficient integrity to produce a monolithic layer on a furnace wall. Because of the heat to which the layer is subjected  
15 incomplete coating of the fibers would result in premature failure of the lining.

          In practice, the method is best carried out by adding water to the tacky high temperature inorganic binder concentrate in a large drum located at the site of the surface to be sprayed. Bulk,  
20 high temperature resistant fibers such as alumina-silica-zirconia fibers are placed in a hopper and may be shredded if needed to allow blowing thereof through spray gun 14. The liquid binder concentrate is mixed with water in the drum preferably in approximately the ratio of, by volume, 4.5 parts water to 1 part binder concentrate.  
25 It should be expressly understood that the dilution ratio depends upon binder concentration and specific applications. Spraying the diluted binder directly onto the surface to be coated improves the bond of the fiber binder layer to the surface. The diluted binder is constantly stirred in the drum as it is fed to the spray gun in  
30 order to ensure a homogenous mixture of diluted binder. Referring to Fig. 6, the diluted binder solution is fed through binder feed line 28 into the external nozzle manifold 26 which feeds nozzles 31. Binder is supplied to nozzle 36 through line 35. Nozzles 31 and 36 spray binder into fiber stream 24. A ratio of about 1.75 lbs.  
35 fiber to about 1 lb. liquid binder is preferred. As with binder dilution, this ratio may be varied according to specific binder dilutions and application. .

The method of the present invention ensures uniform coating of the fibers while in transit to the surface to be sprayed such that the fibers will adhere to each other as well as to the surface and form layers of wetted fibers which may be built up to the desired thickness. In practice, thicknesses of up to 14 inches of wetted fiber have been achieved. Spray gun 14 may be located between about two to four feet or more from the surface as desired. The sprayed binder solution may converge along the axis of the sprayed fibers, either before reaching the surface or, in some cases, the point of convergence may not occur at all before reaching the surface but may be at an imaginary point beyond the surface to be sprayed. The bulk fiber may be blown by a suitable blowing apparatus (not shown), preferably at a rate of up to about 200 cubic feet of air per minute through the fiber feed line 23.

While water is the preferred diluent because of its availability and cost, other suitable inert diluents such as alcohol or ethers may be used. Once the coated fibers have been applied to the surface as in Fig. 1, the layer of binder coated fibers may be tamped as by the second operator 21 in Fig. 1. Operator 21 is shown utilizing a compressing trowel 22 in order to pack the coated fibers to the desired density. Once the fibers have been applied to the surface to the desired density, the binder is cured, by raising the temperature of the furnace to at least 350°F and preferably to about 450°F or more. While this is the preferred step for curing the fibers, any curing method which drives off the water or other diluent and all moisture from the coated fiber layer may be utilized with the method of the present invention.

As shown in Fig. 1, the method of the present invention may be used to apply binder coated fiber to a variety of surfaces. While the stream of binder coated fiber may be applied directly to the surfaces without support, it may be desirable in some instances to provide additional support for the fiber layer. That is, for example, expanded metal lathe could be attached to ceiling 17 of furnace 11 in order to provide additional support for the binder coated fiber layer. Any other supporting structure as is known in the art may also be utilized. Additionally, it should be noted that the binder coated fiber is preferably applied to a surface which has

been properly prepared. That is, in the case of refractory brick or other monolithic dense refractory such as shown on wall 18, such brick should be sand-blasted or otherwise prepared to remove loose or flaky material from the surface to be coated. If the binder coated fiber is to be applied to a metal surface as in 17 or wall 16 in Fig. 1, a asphaltum or other protective coating may be desired in some cases. Where the method is to be used to apply binder coated fiber to a wall of existing refractory fiber as in wall 19 of Fig. 1, it is desirable to again remove all loose or flaking surfaces from the surface to be coated. The binder coated fibers may also be applied over an existing layer of previously sprayed binder coated fibers or fibrous formed shapes. That is, should a portion of the previous layer be knocked off due to mechanical or other contact with the surface, a new layer of binder coated fibers may be applied over the damaged area. In many situations it is desirable to spray a coating of binder onto the fiber-binder layer after the layer has been applied to the surface.

It can readily be seen that this method of applying refractory fiber linings overcomes many of the shortcomings previously described. This method, for instance, does not require the application of a mortar to old brick work or refractory linings in order to apply fibers in modular form as in prior art methods. The binder supplies the adhering mechanism as the new layer is being applied which results in reduced labor costs. Nor does the present method require any particular waiting period for the mortar to form hydraulic bonds and then slowly dry out prior to firing. It can also be seen that the present method eliminates one of the basic shortcomings in prior attachment methods which required the exposed refractory fiber layer to be coated with a bonding material after the layers or modules were installed to impart abrasion resistance. The present method ensures that all fibers throughout the layer are well bonded to one another forming an inherently superior monolithic lining.

The surface of the lining applied with the present method is far less affected by mechanical or gas velocity abrasion. To support this statement the following experiment was conducted: a layer approximately 2 inches thick of 2600°F refractory fiber

lanville CERACHROME) was sprayed according to the present method onto an 12" x 9" free standing refractory brick wall. This wall was placed directly in front of an air natural gas burner port of a furnace so that the surface of the sprayed lining was at 90°F to the flame at a distance of 24" from the burner. Upon repeated firing to temperatures of 2200°F, 2400°F, and 2600°F, there was no damage to the sprayed layer from this extreme condition of temperature and gas stream velocity.

10 A novel method has been disclosed for applying refractory or other fiber which has been coated with a binder to a surface. Because the method ensures bonding together of the high temperature resistant fibers and their adherence to a surface, thick layers of heat insulating fibers may be applied very quickly. Because the  
15 the time normally required by present methods, the cost of the installation as well as the down time of the furnace or other device to be insulated are substantially reduced. Finally, the method disclosed herein allows immediate heating of the fiber layer thereby allowing the furnace or other device to be put into service almost  
20 immediately after spraying. In fact, with the use of remote controlled devices such as robots or an air on water cooled lance, the binder fiber layer could be applied to a furnace which is at operating temperature. This would be useful to repair furnaces while still in operation.

25 In accordance with the present invention, an excellent binder system has been discovered which preferably comprises chromium aluminum phosphate and a suitable tackifying agent. The chromium aluminum phosphate compound preferred in the present invention may be characterized by the following generalized  
30 formula:  $Al_2O_3 \cdot 3P_2O_5 \cdot xCr_2O_3$  wherein x ranges from about 0.1 to 10.

The term "tackifying agent", as used with respect to the present invention, is meant to define those substances which impart tackiness or adhesive properties to the binder system of the present  
35 invention. Generally, such tackifying agents will be inorganic in nature. High temperature resistant clays, especially montmorillonite clays (preferably the western bentonite variety)

have been found to be useful in the present invention. Sodium and calcium based clays such as southern bentonite may also be used as a tackifying agent. Whatever tackifying agent is used should, of course, be compatible with the overall binder system and its intended use with regard to both chemical and physical properties. It is especially important that the tackifier, in the quantity used, not produce fluxing of the binder-fiber system at elevated temperatures.

While the inventive binder system may be made according to any suitable method known to those skilled in the art, the superior binder system of the present invention is preferably made according to the generalized process described herein below.

A mixture of phosphoric acid solution, an appropriate fluxing agent such as boric acid, and bentonite clay are stirred together and then heated to a temperature (typically 100°F) sufficient for the ingredients to react with one another. Although varying concentrations of phosphoric acid may be used, in the present invention a 75% or higher phosphoric acid solution is preferred. As used herein, the term appropriate fluxing agent is meant to include those substances which will impart added strength to the inventive binder system by improving glassy bonding at elevated temperatures. While boric acid is a preferred fluxing agent, other inorganic metal salts such as sodium carbonate, magnesium chloride, magnesium nitrate, calcium carbonate, cobalt oxide, and others may be employed.

After heating to approximately 100°F the above reaction mixture is combined with a hydrated alumina. The preferred alumina is a hydrated alumina such as that currently marketed by ALCOA under the trademark C-31. The mixture is then heated to a temperature of approximately 180°F at which time chromic oxide ( $\text{Cr}_2\text{O}_3$ ) is added. The preferred chromic oxide has a specific gravity of 5.1 and is added in a percent, by weight, of 1.26% of the total mixture weight. At this point the reaction becomes exothermic, the temperature rises to approximately 233°F, the volume of the mixture approximately doubles, and the reaction is complete. The solution is allowed to cool and an inert carrier such as water is added to adjust the specific gravity of the binder to 1.70 at room temperature.

The above ingredients are used in the present invention at the following general, preferred, and most preferred weight percentage levels based upon the total weight of the binder.

5

TABLE

	<u>Ingredient</u>	<u>General</u>	<u>Preferred</u>	<u>Most Preferred</u>
10	Phosphoric Acid Solution	50-90	75-85	78.8
	Fluxing Agent	0-5	2-4	2.37
15	Tackifying Agent	1-3	1-5	2.87
	Hydrated Alumina	5-30	10-20	14.2
20	Chromium Compound	0.5-5	1-2	1.26

25 While chromic oxide  $\text{Cr}_2\text{O}_3$  has been disclosed, other suitable chromium compounds include solutions of chromic acid ( $\text{H}_2\text{CrO}_4$ ), and chromium salt solutions such as magnesium chromate, which can be converted under appropriate temperature conditions to chromia ( $\text{Cr}_2\text{O}_3$ ), may be added.

30 Similarly, while alumina has been disclosed as the preferred metal oxide to be reacted with the phosphoric acid solution, magnesium oxide or other suitable metal oxides may be reacted with the phosphoric acid solution without departing from the scope of the invention.

35 An alternate binder formulation includes colloidal silica combined with a tackifying agent. Preferably NALCO 1115 Colloidal Silica of the 4 millimicron size Sol containing 15% solids is used. Approximately forty pounds (40 lbs) of this colloidal silica was mixed with 1.25 pounds of western bentonite to form a tacky binder.

5 The addition of a montmorillonite clay such as western bentonite to the binder matrix has been found to produce a tacky binder which adheres to virtually any surface. While Western Bentonite is preferred, any of the clays selected from the Montmorillonite types of clay minerals have been found to be suitable tackifying agents.

10 The binder of the present invention may be advantageously employed with refractory fibers in a spray-on process. That is, refractory fibers may be directed from a spray gun while at the same time being coated with the binder of the present invention. Such application of refractory fiber and binder has been found to raise the operating temperature of the refractory fibers above their normal rated temperature. For application of refractory fiber to a surface such as a furnace wall, the binder is diluted, preferably  
15 with water, in a 4.5 to 1 water to binder ratio by volume. This ratio may range from 2 to 1 to 15 to 1 by volume. The diluted binder is preferably stirred in the dilution container to ensure a homogenous mixture during the spraying process. The diluted binder may be sprayed with the fiber onto a surface in a ratio which  
20 depends upon binder dilution. For the preferred chromium aluminum phosphate binder disclosed above, a ratio of 1.75 lbs. of fiber to 1.0 lb. binder is preferred.

Examples of different binder formulations and concentrations are listed below:

25 Binder #1 was a mixture of NALCO AG 1115 colloidal silica marketed by NALCO Chemical Corp. of 4 millimicron size particles containing 15% solids. Forty pounds of this solution was mixed with 1.25 lbs. of western bentonite. The resulting binder was sprayed in a 30% binder 70% alumina-silica-zirconia fiber ratio by weight and  
30 cured at 1000°F. A 12 lb/ft<sup>3</sup> composite material resulted having good integrity.

Binder #2 was a mixture of the chromium aluminum phosphate binder matrix with bentonite clay and boric acid in the most preferred formulation previously disclosed. This binder concentrate  
35 was diluted 4 to 1 by volume with water and sprayed with alumina-silica-zirconia fiber in a ratio of 14% binder to 86% fiber by weight. This composite was then cured at 1000°F and produced a 12 lb/ft<sup>3</sup> material.

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Binder #3 was a mixture of 1/2 lb. colloidal alumina, 2 lbs. of -325 mesh  $Al_2O_3$  particles and 1 1/4 lbs. bentonite in 40 lbs. of water. This binder concentrate was sprayed with alumina-silica-zirconia fibers in a ratio of 40% binder to 60% fiber. The resulting material was cured at 1000°F and produced a material which did not adhere well to the brick substrate and did not produce material of sufficient integrity to allow density to be accurately measured.

Binder #4 was a mixture of 11 lbs. of Kaolin Clay, 3/4 lbs. western bentonite and 30 lbs. of water. This binder mixture was too viscous to be sprayed.

Binder #5 was a mixture of the chromium aluminum phosphate binder matrix with bentonite clay and boric acid in the most preferred formulation previously disclosed. This binder concentrate was diluted 10 to 1 by volume with water and sprayed in equal amounts by weight with alumina-silica-zirconia fiber. The resulting material was cured at 1000°F and produced a material of 15 lb/ft<sup>3</sup> density.

Binder #6 was a mixture of the chromium aluminum phosphate binder matrix with the bentonite clay and boric acid in the most preferred formulation previously disclosed. This binder concentrate was diluted 15 to 1 by volume with water and sprayed in a ratio of 45% binder to 55% alumina-silica-zirconia fiber by weight. The resulting material was cured at room temperature and produced a material of 13 lbs/ft<sup>3</sup> density.

Binder #7 was a mixture of the chromium aluminum phosphate binder matrix with bentonite clay and boric acid in the most preferred formulation previously disclosed. This binder was diluted 15 to 1 by volume with water and sprayed in a ratio of 36% binder to 64% alumina-silica-zirconia fiber by weight. The resulting material was cured at 1000°F but did not produce a material of sufficient integrity to obtain a density measurement.

Binder #8 was the colloidal silica binder disclosed as binder #1 without the bentonite clay. The binder was sprayed in a 60% binder to 40% fiber ratio by weight with alumina-silica-zirconia fibers. The resulting material was cured at

1000°F but did not produce a material of sufficient integrity to obtain a density measurement due to binder migration.

Binder #9 was a mixture of bentonite clay and water in a ratio by weight of 10% bentonite to 90 % water. This binder did not bond with the alumina-silica-zirconia fiber.

A comparison of characteristics of the binder formulations is given below:

		Binder <u>Tacky</u>	Integrity with Fibers after <u>Curing</u>	Binder <u>Migration</u>
10	Binder #1	Yes	Good	Some
15	Binder #2	Yes	Fair	Very little
	Binder #3	Yes	Fair	Some
20	Binder #4	Yes	Unknown	Unknown
	Binder #5	Yes	Excellent	Very little
25	Binder #6	Yes	Excellent	Very little
	Binder #7	Yes	Fair	Unknown
30	Binder #8	No	Poor	Very heavy
	Binder #9	Yes	None	Unknown

35 A comparison of shrinkage between the materials of binders #1, 2, 3, 5, and 6 was made with each other and with the fiber alone. After being exposed to a temperature of 2400°F for four

hours the binder - fiber composites exhibited the following shrinkage characteristics:

	<u>Shrinkage (%)</u>
Binder #1	2.6
Binder #2	1.5
Binder #3	1.6
Binder #5	1.6
Binder #6	1.4
Fiber alone	2.0

10

From the above it can be seen that composites formed from the most preferred binder concentrate formulation of binder #2, 5 and 6 exhibited less shrinkage than the fiber alone. While binder #3 exhibited low shrinkage, the specific binder formulation exhibited only fair bonding characteristics.

A comparison of curing temperatures was made for binder #5. That is, the material made under binder formulation #5 was cured at 250°F, 350°F and 450°F. Each material was then placed in a 90% humidity chamber for 72 hours with the following results: the material cured at 250°F received 31% added moisture and was wet and soft suggesting incomplete curing; the material cured at 350°F received 12% added moisture and was somewhat wet and soft, but was marginally acceptable; and the material cured at 450°F received 13% added moisture and was hard and held its integrity. Thus the curing temperature has a lower limit of about 350°F with a preferred curing temperature of about 450°F or more.

In order to determine the preferred limits of dilution and binder to fiber ratio for the most preferred chromium aluminum phosphate binder, the formulations of binders #2, 5, 6 and 7 were tested. The fiber-binder composite materials were compared with respect to the weight ratio of the original binder concentrate to fiber weight ratios in light of the integrity observed in the resulting material. The ratio of the weight of binder concentrate to fiber was 4.86% for binder #2; 14.52% for binder #5; 8.33% for binder #6; and 5.72% for binder #7. The materials produced by binders #5 and 6 had excellent integrity while the material from binders #2 and 7 had fair integrity. This suggests that the

preferred binder concentrate to fiber weight ratio should be at least 6%. Thus, the binder dilution and diluted binder to fiber weight ratio should be adjusted accordingly.

5 While the invention has been disclosed with respect to preferred embodiment thereof, it is not to be so limited as changes and modifications may be made which are within the full intended scope of the invention as defined by the appended claims. For example, as shown in Figs. 2 and 5, various geometric configurations of the spray gun may be employed so as to direct the path of the  
10 fibers to a specific application. In addition, various diluents may be used as the carrier for the binder concentrate. While chromium aluminum phosphate with bentonite clay has been disclosed as the preferred binder, other binders employing a temperature resistant clay such as montmorillonite, or plastic ball clays containing  
15 montmorillonite clay in quantities sufficient to ensure a tacky inorganic binder, may be employed. Finally, while the inventive method is disclosed with reference to spraying refractory fibers, any fiber such as fiberglass, mineral wool, or other suitable fibers may be utilized with the disclosed method. Combinations of these or  
20 different refractory fibers could also be employed to produce a stratified furnace lining which is extremely cost effective. In addition, the spraying of a binder with particulate matter such as various metallic oxides could be employed to provide a particularly heat resistant surface over the sprayed fiber-binder layer.

Claims

What is claimed is:

1. A method for applying a layer of fibers to a surface comprising the steps of:

5 directing a stream of said fibers toward said surface;  
coating said directed fibers with a tacky inorganic  
binder during said step of directing such that  
said fibers adhere to one another and to said  
surface; and  
10 curing said coated fibers.

2. Method according to Claim 1 wherein said fibers include refractory fibers.

3. Method according to Claim 1 further including the step of diluting said inorganic binder prior to said step of directing.

15 4. Method according to Claim 1 further including the step of compressing said binder coated fiber layer to a desired density prior to said step of curing.

5. Method according to Claim 1 wherein said step of curing includes exposing a face of said fiber layer opposite to said  
20 surface to a temperature of at least 350°F.

6. Method of Claim 1 wherein said step of coating said fibers occurs as said fibers are directed from a spray gun.

7. Method according to Claim 1 wherein said binder is sprayed into said fiber stream.

25 8. Method according to Claim 1 wherein said binder includes a suspension of colloidal particles, the oxide of which will not act as a fluxing agent to alumina silica fibers at temperature above 1500°F.

9. Method according to Claim 1 wherein said binder  
30 includes a phosphate compound and montmorillonite clay.

10. Method according to Claim 1 further including the step of spraying said binder onto said surface prior to said step of directing.

35 11. A method for producing a layer of fibers comprising the steps of:

directing said fibers in a stream configuration;  
directing a tacky inorganic binder into said stream  
configuration so as to coat said fibers with said  
binder causing said fibers to adhere to one  
another; and

curing said coated fibers.

12. Method according to claim 1 further including the step  
of, prior to curing said coated fibers:

directing a stream of particulate material such as a  
metal oxide toward said coated fibers; and  
coating said particulate material with said binder  
during said step of directing such that said  
coated particulate material adheres to one  
another and to said coated fibers.

13. A refractory binder comprising:  
a phosphate matrix; and  
a tackifying agent in intimate admixture with said  
phosphate matrix.

14. Binder according to Claim 13 wherein said tackifying  
agent is montmorillonite clay.

15. Binder according to Claim 13 wherein said tackifying  
agent is bentonite clay.

16. Binder according to Claim 13 further including a  
fluxing agent.

17. Binder according to Claim 13 further including a metal  
oxide selected from the group consisting of alumina, magnesium or  
silica.

18. Binder according to Claim 16 wherein said fluxing  
agent is boric acid.

19. Binder according to Claim 13 further including a  
chromium compound.

20. Binder according to Claim 19 wherein said chromium  
compound is chromic oxide.

21. A refractory binder comprising by weight percent:

chromium compound	1- 2%
phosphoric acid	75-85%
hydrated alumina	10-20%
tackifying agent	1- 5%

22. A refractory binder according to Claim 21 further including, up to 5% by weight percent of a fluxing agent.

23. A refractory binder according to Claim 21 wherein said tackifying agent includes montmorillonite clay.

5           24. A refractory binder comprising:  
            a suspension of colloidal particles the oxide of which  
                will not act as a fluxing agent to alumina silica  
                fibers at temperatures above 1500°F; and  
            a tackifying agent in intimate admixture with the said  
10           suspension.

25. Binder according to Claim 24 wherein said colloidal suspension particles are selected from a group consisting of alumina, silica, or zirconia.

15           26. Binder according to Claim 24 wherein said tackifying agent includes a montmorillonite clay.

27. Method for making a refractory binder comprising the steps of:

            stirring a phosphate - tackifying agent mixture;  
            heating said mixture to at least approximately 100°F;  
20           adding hydrated alumina to said mixture; and  
            heating said hydrated alumina-phosphate-tackifying  
            agent to at least approximately 180°F.

28. Method according to Claim 27 further including the step of adding a chromium compound to said mixture.

25           29. Method according to Claim 27 further including the step of adding a fluxing agent to said phosphate-tackifying mixture.

30. Method according to Claim 27 wherein said tackifying agent includes montmorillonite clay.

31. Method according to Claim 29 wherein said fluxing  
20           agent is boric acid.

32. Method for making a refractory binder comprising the step of reacting a colloidal suspension of particles selected from among the group consisting of alumina, silica or zirconia with a tackifying agent.

35           33. Method according to Claim 32 wherein said tackifying agent is montmorillonite clay.



34. A refractory material comprising:  
a plurality of refractory fibers; and  
a phosphate-tackifying agent mixture.

5 35. Material according to Claim 34 wherein said  
tackifying agent includes montmorillonite clay.

36. Material according to Claim 34 wherein said mixture  
further includes a chromium compound.

37. Material according to Claim 34 wherein said mixture  
further includes a fluxing agent.

10 38. Material according to Claim 37 wherein said fluxing  
agent is boric acid.

39. Material according to Claim 34 wherein said mixture  
further includes alumina.

15 40. A refractory material comprising:  
a plurality of refractory fibers; and  
a binder including:

20 a suspension of colloidal particles the oxide of  
which will not act as a fluxing agent to said  
refractory fibers at temperatures above 1500°F;  
and  
a tackifying agent in intimate admixture with  
said suspension.

25 41. Material according to Claim 40 wherein said colloidal  
suspension particles are selected from a group consisting of  
alumina, silica, or zirconia.

42. Material according to Claim 40 wherein said tackifying  
agent includes montmorillonite clay.

30 43. In a spray gun for directing a stream of moving  
material including a feed line for confining said stream of moving  
material in a predetermined configuration and at least one outside  
spray nozzle located on said spray gun for directing a stream of  
binder onto the outside of said stream of moving material, the  
improvement comprising:

35 at least one inside spray nozzle located within said moving  
stream.

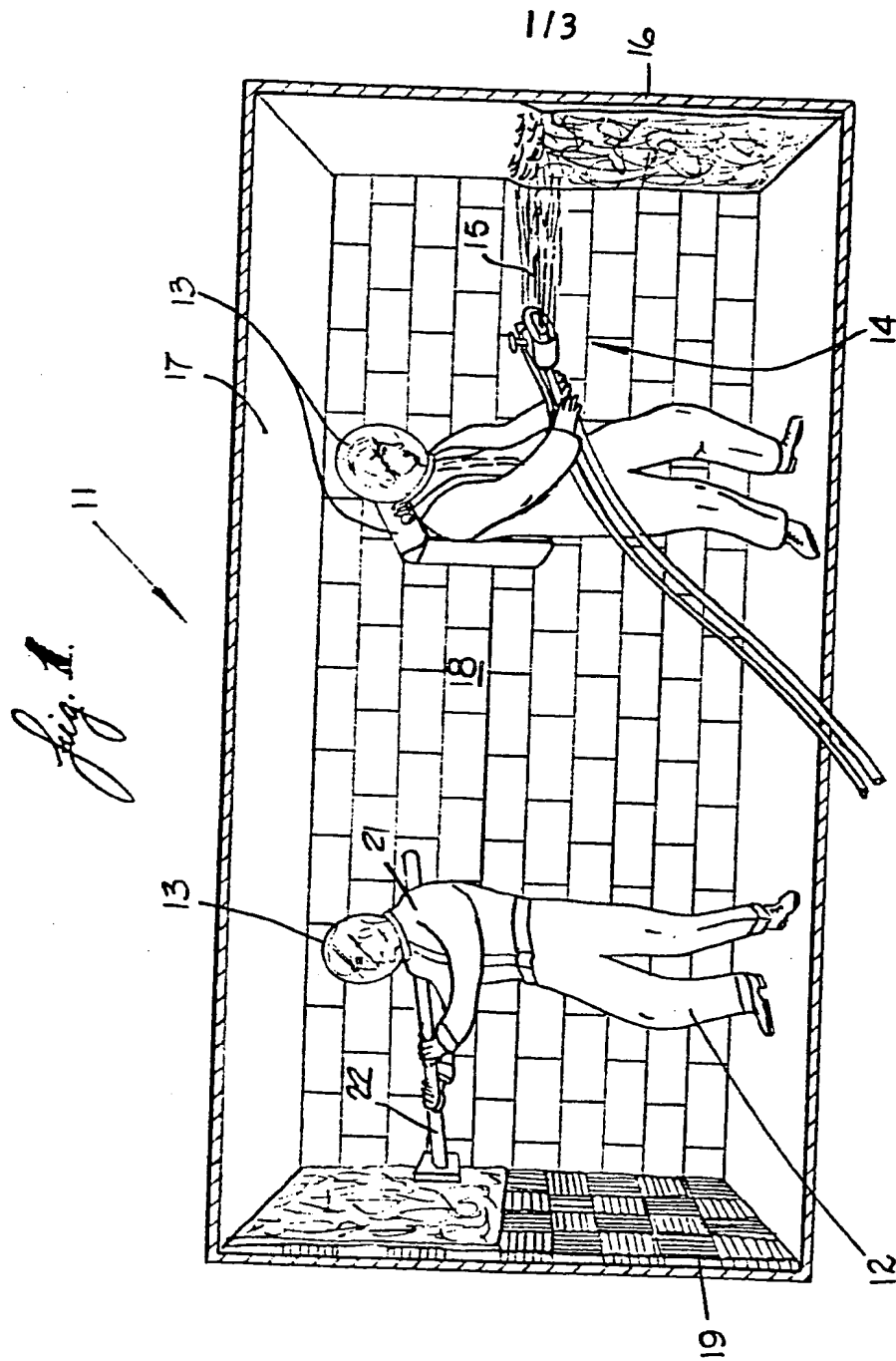
44. A spray gun comprising:

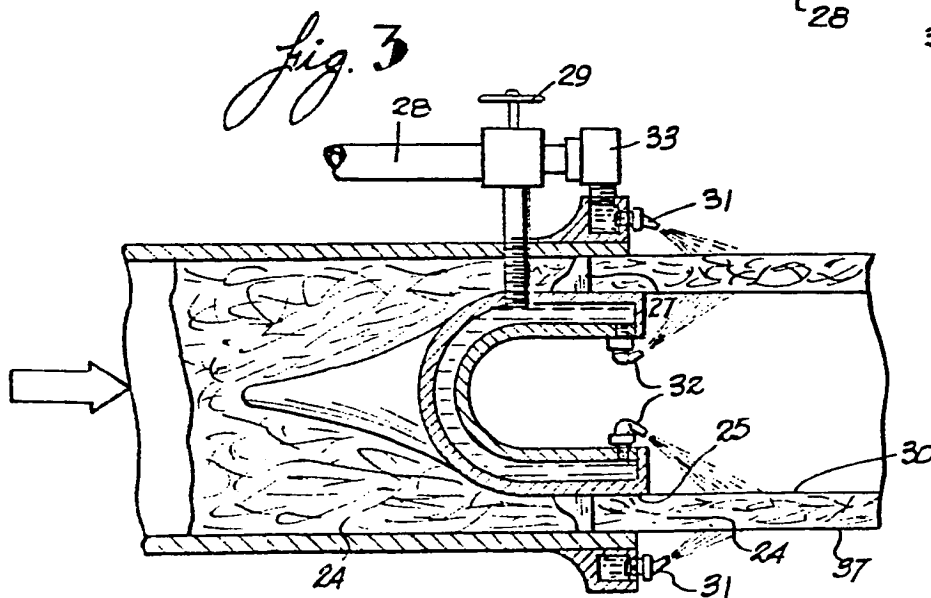
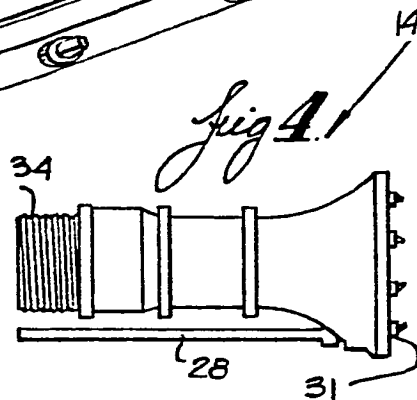
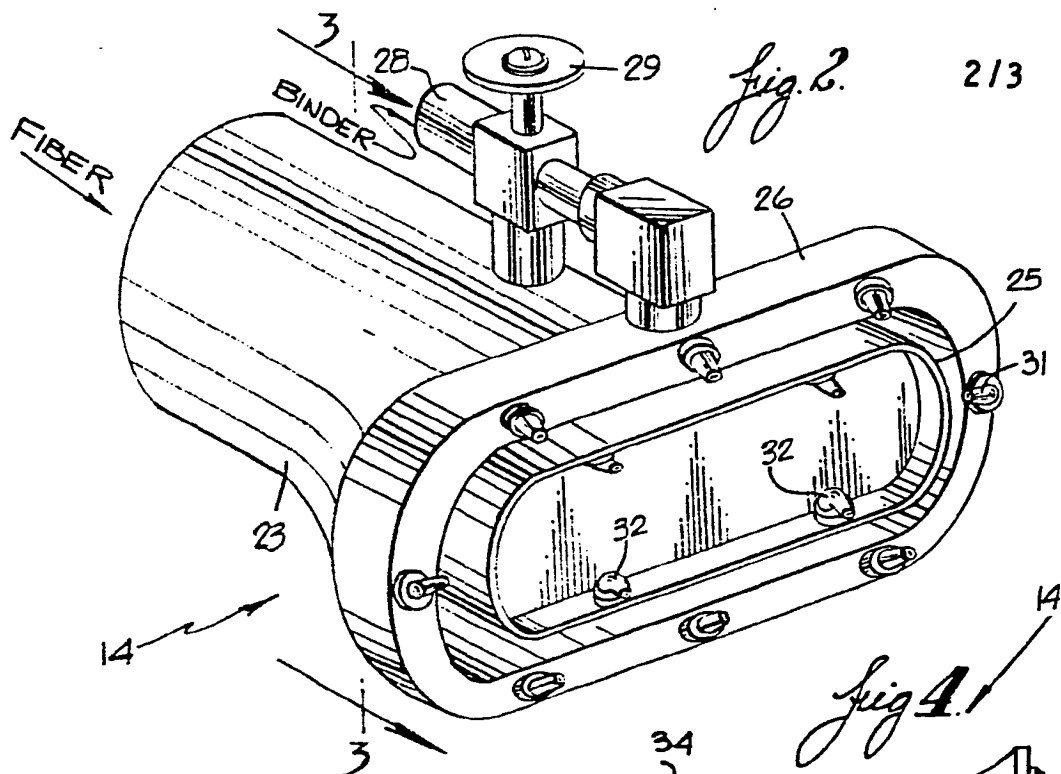
a feed line for directing a stream of moving material in a predetermined path;

a deflector located within said feed line for configuring said moving stream of material in an annular path;

at least one spray nozzle connected to said spray gun outside of said annular path; and

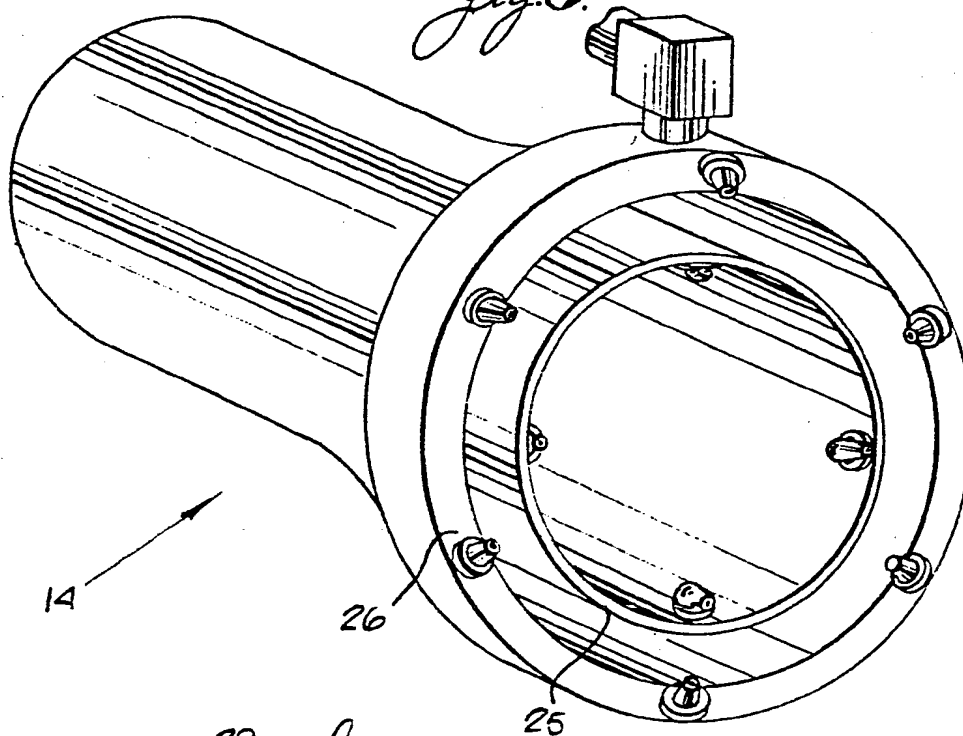
at least one spray nozzle located on said spray gun inside of said annular path.



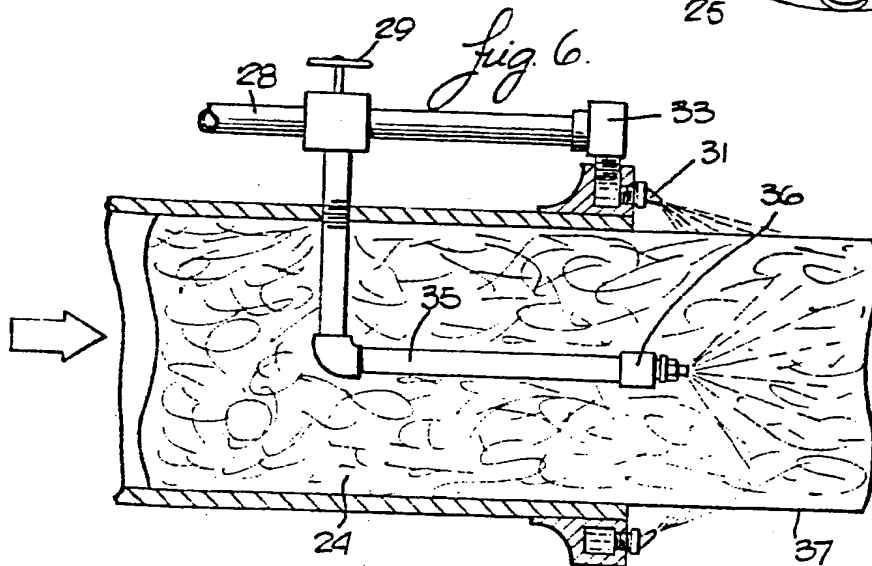


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*fig. 5.*



*fig. 6.*



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## EUROPEAN PATENT APPLICATION

21 Application number: 84112530.5

22 Date of filing: 17.10.84

51 Int. Cl.<sup>4</sup>: F 24 D 1/00  
B 05 B 7/06, F 23 M 5/00  
C 04 B 28/34  
/(C04B28/34, 14:38)

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54 Insulation system.

67 A method and apparatus for directing a stream of bulk fibers while simultaneously spraying an inorganic temperature resistant tacky binder material into the stream of fibers. A layer of high temperature insulating fibers may thus be formed. The binder may be a phosphate tackifying agent mixture or a colloidal suspension of materials with the tackifying agent. The tackifying agent is preferably montmorillonite clay.

EP 0 142 715 A3



European Patent  
Office

# EUROPEAN SEARCH REPORT

0142715

Application number

EP 84 11 2530

## DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,Y	US - A - 2 929 436 (W.J.HAMPSHIRE)		F 24 D 1/00
A	* Claims 1,2 *	1,11	B 05 B 7/06
A	* Figure 2 *	6,7 43	F 23 M 5/00
	--		C 04 B 28/24//
			(C 04 B 28/34
			C 04 B 14/38)
Y	DE - A - 1 458 912 (PURMETALL)		
A	* Page 3, lines 15-20; claim 1 *	1,11	
A	* Claim 2 *	3	
	* Claim 4 *	9	
	--		
A	US - A - 3 674 599 (A.J.WILTSHIRE)		
	* Claim 1; figure 1 *	4	
	--		
Y	US - A - 2 433 463 (A.J. LAMPE)		
	* Column 1, lines 10-22; column 2, lines 4-7; figures *	43,44	
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Y	FR - A - 1 211 519 (CIMEX TRUST)		
	* Page 6, left-hand column, lines 9-26; right-hand column, lines 8-12; figures 10,11,12 *	43,44	
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X	GB - A - 2 093 014 (DIDIER-WERKE A.G.)		
	* Page 3, lines 18-25; examples 1-3; claims 1,2 *	13-15, 17,19-23	
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
The Hague		10-06-1985	VAN THIELEN
CATEGORY OF CITED DOCUMENTS			
C : particularly relevant if taken alone		T : theory or principle underlying the invention	
Y : particularly relevant if combined with another document of the same category		E : earlier patent document, but published on, or after the filing date	
A : technological background		D : document cited in the application	
O : non-written disclosure		L : document cited for other reasons	
P : intermediate document		& : member of the same patent family, corresponding document	

EP 0 Form 1503 03 82





**CLAIMS INCURRING FEES**

The present European patent application comprised at the time of filing more than ten claims.

- ☐ All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid, namely claims.
- ☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

**X LACK OF UNITY OF INVENTION**

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely:

- 1) Claims 1-12: Method for applying layer of fibres and 43-44 apparatus therefore
- 2) Claims 13-23: Refracting binder "A", 27-30 method for making binder "A", 34-39 use of binder "A"
- 3) Claims 24-26: Refractory binder "B", 32-33 method for making binder "B", 40-42 use of binder "B"

- ☒ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:
- ☐ None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:



DOCUMENTS CONSIDERED TO BE RELEVANT																	
Category	Citation of document with indication where appropriate of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)														
X	CHEMICAL ABSTRACTS, vol. 89, no. 16 1978, abstract 134484G, page 311 Columbus, Ohio, US & JP - A - 78 46 324 (NIPPON AS- BESTOS CO. LTD) (25-04-1978)  * The whole abstract *	24-26, 32,33 40-42															
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X	CHEMICAL ABSTRACTS, vol. 84, no.12, 1976, abstract 76925J, page 331, Columbus, Ohio, US & JP - A - 75 141 618 (NISSAN CHE- MICAL INDUSTRIES LTD) (14-11-1975)  * The whole abstract *	24-26 32,33, 40-42															
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X	CHEMICAL ABSTRACTS, vol. 82, no.26, 1975, abstract 172760G, page 102 Columbus, Ohio, US & JP - A - 74 122 539 (KIKUSUI KAGAKU KOGYO CO LTD) (22-11-1974)  * The whole abstract *	24-26, 32,33	TECHNICAL FIELDS SEARCHED (Int. Cl. 4)														
A	CHEMICAL ABSTRACTS, vol. 98, no. 4, 1983, abstract 21424E, page 283 Columbus, Ohio, US & JP - A - 57 106 771 (NISSAN CHEMICAL INDUSTRIES LTD) (02-07- 1982)  * The whole abstract *	40-42															
The present search report has been drawn up for all claims																	
Place of search		Date of completion of the search	Examiner														
<table border="0"><tr><td>CATEGORY OF CITED DOCUMENTS</td><td>T : theory or principle underlying the invention</td></tr><tr><td>X : particularly relevant if taken alone</td><td>E : earlier patent document, but published on, or</td></tr><tr><td>Y : particularly relevant if combined with another</td><td>after the filing date</td></tr><tr><td>document of the same category</td><td>D : document cited in the application</td></tr><tr><td>A : technological background</td><td>L : document cited for other reasons</td></tr><tr><td>O : non-written disclosure</td><td>&amp; : member of the same patent family, corresponding</td></tr><tr><td>P : intermediate document</td><td>document</td></tr></table>				CATEGORY OF CITED DOCUMENTS	T : theory or principle underlying the invention	X : particularly relevant if taken alone	E : earlier patent document, but published on, or	Y : particularly relevant if combined with another	after the filing date	document of the same category	D : document cited in the application	A : technological background	L : document cited for other reasons	O : non-written disclosure	& : member of the same patent family, corresponding	P : intermediate document	document
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